

Bis[bis(pentamethylcyclopentadienyl)-cobalt(III)] tetrachloridocobaltate(II) dichloromethane disolvate

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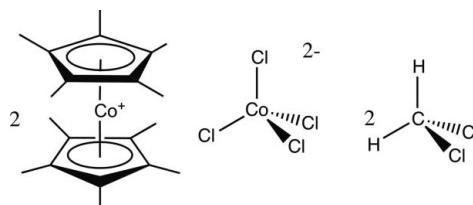
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Key indicators: single-crystal X-ray study; $T = 101\text{ K}$; mean $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$; disorder in solvent or counterion; R factor = 0.037; wR factor = 0.091; data-to-parameter ratio = 32.0.

The title compound, $[\text{Co}(\text{C}_{10}\text{H}_{15})_2]_2[\text{CoCl}_4]\cdot 2\text{CH}_2\text{Cl}_2$, was isolated as a dichloromethane solvate and was formed in the reaction between lithium pentamethylcyclopentadienide and anhydrous cobalt(II) chloride in tetrahydrofuran. There are two decamethylcobaltocenium cations, one tetrachloridocobaltate(II) anion and two dichloromethane solvent molecules in the formula unit. There is a slight disorder of the dichloromethane solvent which was treated with a two-site model [occupancy rates = 0.765 (4) and 0.235 (4)]. The dichloromethane molecules display significant $\text{C}-\text{H}\cdots\text{Cl}$ interactions with the tetrachloridocobaltate(II) dianion. The cobalt atom of the decamethylcobaltocenium cation sits on a twofold rotation axis, with only one pentamethylcyclopentadiene ligand being unique and the second generated by symmetry. The cobalt atom of the $[\text{CoCl}_4]^{2-}$ ion sits on a special site with $\bar{4}$ symmetry, with one unique chloride ligand and the others generated by the fourfold inversion axis.

Related literature

For a related structure with a $(\text{THF})_2\text{LiCl}_2\text{CoCl}_2$ monoanion and the decamethylcobaltocenium cation, see: Dehnen & Zimmermann (2000) (CCDC 135478). The structure of a related dimer synthesized by Koelle *et al.* (1986) was determined by Olson & Dahl (1986) (CCDC 566220). For a discussion of the role of chloroform and dichloromethane solvent molecules in crystal packing, see: Allen *et al.* (2013).



Experimental

Crystal data

| | |
|---|--|
| $[\text{Co}(\text{C}_{10}\text{H}_{15})_2]_2[\text{CoCl}_4]\cdot 2\text{CH}_2\text{Cl}_2$ | $Z = 2$ |
| $M_r = 1033.35$ | $\text{Mo } K\alpha$ radiation |
| Tetragonal, $P4_2/n$ | $\mu = 1.48\text{ mm}^{-1}$ |
| $a = 12.20980 (12)\text{ \AA}$ | $T = 101\text{ K}$ |
| $c = 16.2811 (3)\text{ \AA}$ | $0.27 \times 0.24 \times 0.18\text{ mm}$ |
| $V = 2427.17 (7)\text{ \AA}^3$ | |

Data collection

| | |
|--|--|
| Agilent Xcalibur Gemini Ultra diffractometer with Eos detector | 26858 measured reflections |
| Absorption correction: gaussian (<i>CrysAlis PRO</i> ; Agilent, 2013) | 4161 independent reflections |
| $T_{\min} = 0.755$, $T_{\max} = 0.821$ | 3394 reflections with $I > 2\sigma(I)$ |
| | $R_{\text{int}} = 0.037$ |

Refinement

| | |
|---------------------------------|---|
| $R[F^2 > 2\sigma(F^2)] = 0.037$ | 130 parameters |
| $wR(F^2) = 0.091$ | H-atom parameters constrained |
| $S = 1.04$ | $\Delta\rho_{\max} = 1.21\text{ e \AA}^{-3}$ |
| 4161 reflections | $\Delta\rho_{\min} = -1.07\text{ e \AA}^{-3}$ |

Table 1
Hydrogen-bond geometry (\AA , $^\circ$).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|---|--------------|--------------------|-------------|----------------------|
| $\text{C11}-\text{H}\cdots\text{Cl1}^{\text{i}}$ | 0.97 | 2.71 | 3.548 (3) | 145 |
| $\text{C11}-\text{H}\cdots\text{Cl1}^{\text{ii}}$ | 0.97 | 2.71 | 3.548 (3) | 145 |

Symmetry codes: (i) $-y + \frac{3}{2}, x - 1, -z + \frac{1}{2}$; (ii) $y + 1, -x + \frac{3}{2}, -z + \frac{1}{2}$.

Data collection: *CrysAlis PRO* (Agilent, 2013); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2008); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZL2564).

References

- Agilent (2013). *CrysAlis PRO*. Agilent Technologies UK Ltd, Yarnton, Oxfordshire, England.
- Allen, F. H., Wood, P. A. & Galek, P. T. A. (2013). *Acta Cryst. B* **69**, 379–388.
- Dehnen, S. & Zimmermann, C. (2000). *Chem. Eur. J.* **6**, 2256–2261.
- Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
- Koelle, U., Fuss, B., Belting, M. & Raabe, E. (1986). *Organometallics*, **5**, 980–987.
- Olson, W. L. & Dahl, L. F. (1986). *Acta Cryst. C* **42**, 541–544.
- Sheldrick, G. M. (2008). *Acta Cryst. A* **64**, 112–122.

supplementary materials

Acta Cryst. (2013). E69, m504 [doi:10.1107/S160053681302254X]

Bis[bis(pentamethylcyclopentadienyl)cobalt(III)] tetrachloridocobaltate(II) di-chloromethane disolvate

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1. Comment

Koelle *et al.* (1986) reported on the preparation of a pentamethylcyclopentadienylcobalt chloro-bridged dimeric compound that represents an excellent precursor to other mononuclear complexes *via* bridge-splitting reactions. There is no doubt that Koelle *et al.* made the described bridged complex since Olson & Dahl (1986) published a crystal structure of the dimer. However, in our laboratories, regardless of the stoichiometry used in attempts to make the chloro bridged dimer described by Koelle *et al.* (1986), the title compound was the only material isolated. The composition of the title compound consists of two decamethylcobaltocenium cations, one tetrachlorocobalt(II) dianion, and two molecules of dichloromethane. The dichloromethane molecules were slightly disordered and the disorder was treated successfully by a two site model with occupancies of 76.5 (4) and 23.5 (4)%. Dehnen & Zimmerman (2000) noted a similar product in attempts to make selenium-bridged compounds and noted the decamethylcobaltocenium ion was formed depending on the temperature of the reaction. In their case, the CoCl_4^{2-} ion was linked *via* chloride bridges to a bis-THF Li^+ cation.

In the structure reported here, the CoCl_4^{2-} ion shows significant interaction with the dichloromethane of solvation. Recently, Allen *et al.* (2013) examined the Cambridge Crystallographic Data Base and analyzed crystallographic evidence of C—H \cdots Cl hydrogen bonding for both CH_2Cl_2 and CHCl_3 . In that paper, for the specific case of CH_2Cl_2 interacting with Cl^- , they note C—H \cdots Cl interactions with H \cdots Cl distances ranging from 2.33 to 2.95 Å and C—H \cdots Cl angles ranging from 120° to 170° for a set of 63 structures. In the title structure, the H \cdots Cl distance is 2.72 Å with a C—H \cdots Cl angle of 145.0°. These parameters would place the C—H \cdots Cl interaction for the title structure very nearly at the median of the structures analyzed by Allen *et al.* (2013).

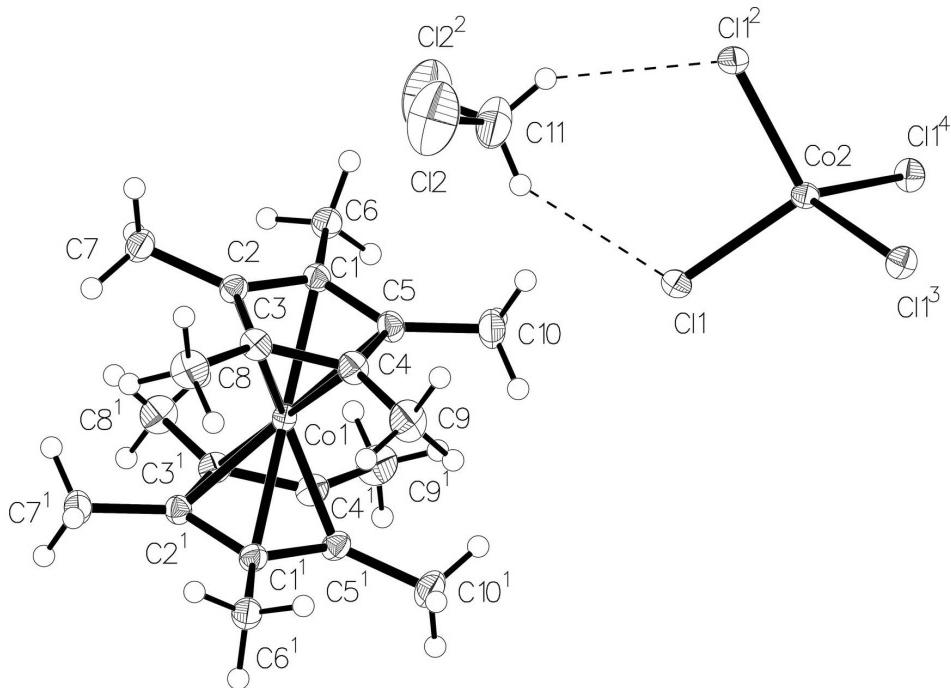
In the structure reported here, the CoCl_4^{2-} ion is also distorted from perfect tetrahedral geometry with the Cl—Co—Cl angles involved in the hydrogen bonding compressed to 100.04 (2)° and the remaining Cl—Co—Cl angles are 114.385 (12)°. While it is important not to make too much of a qualitative observation, the strength of the C—H \cdots Cl interaction may also be responsible for the relative stability of these crystals in open air. Our experience is that, in the absence of a significant attractive interaction, dichloromethane molecules quite easily evaporate from crystals with loss of crystallinity at room temperature.

2. Experimental

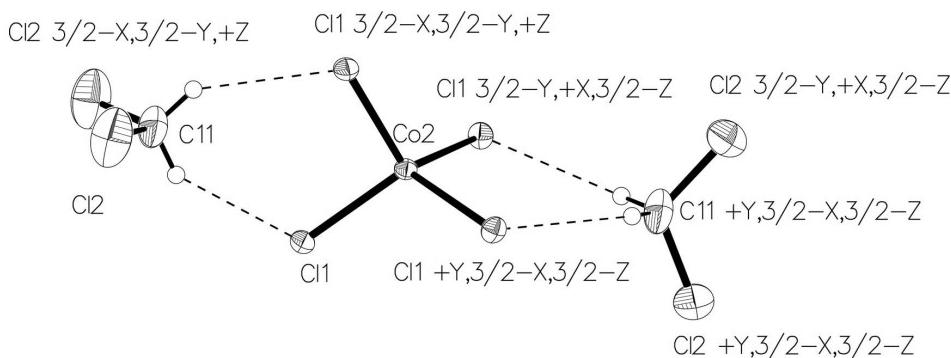
The procedure described by Koelle *et al.* (1986) was followed using lithium pentamethylcyclopentadienide (LiCp^*) and anhydrous cobalt(II) chloride in tetrahydrofuran. Instead of obtaining the hexane soluble brown dimer as described, the reaction produced a green solid. Dissolution of the solid in dichloromethane followed by slow diffusion of diethyl ether produced well formed green prisms of the title compound that are very air stable and retain the dichloromethane of solvation even after several weeks exposure to the open atmosphere at room temperature.

Computing details

Data collection: *CrysAlis PRO* (Agilent, 2013); cell refinement: *CrysAlis PRO* (Agilent, 2013); data reduction: *CrysAlis PRO* (Agilent, 2013); program(s) used to solve structure: *SHELXS* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2008); molecular graphics: OLEX2 (Dolomanov *et al.*, 2009); software used to prepare material for publication: OLEX2 (Dolomanov *et al.*, 2009).

**Figure 1**

Plot of the title compound displaying the complete molecular fragments with thermal ellipsoids shown at 50% probability level. Only the major component (76.3 (4)) of the disordered dichloromethane solvate is shown. Symmetry codes: (1) $3/2 - x, 1/2 - y, z$; (2) $3/2 - x, 3/2 - y, z$; (3) $y, 3/2 - x, 3/2 - z$; (4) $3/2 - y, x, 3/2 - z$

**Figure 2**

Thermal ellipsoid plot of the CoCl_4^{-2} ion showing $\text{C}-\text{H}\cdots\text{Cl}$ interactions with the dichloromethane solvate. Atoms are named with the symmetry operation that generated them. Only the major component (76.3 (4)) of the disordered dichloromethane solvate is shown.

Bis[bis(pentamethylcyclopentadienyl)cobalt(III)] tetrachloridocobaltate(II) dichloromethane disolvate*Crystal data*

$M_r = 1029.32$

Tetragonal, $P4_2/n$

$a = 12.20980 (12) \text{ \AA}$

$c = 16.2811 (3) \text{ \AA}$

$V = 2427.17 (7) \text{ \AA}^3$

$Z = 2$

$F(000) = 1066$

$D_x = 1.408 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.7107 \text{ \AA}$

Cell parameters from 8251 reflections

$\theta = 4.2\text{--}32.2^\circ$

$\mu = 1.48 \text{ mm}^{-1}$

$T = 101 \text{ K}$

Prism, clear green

$0.27 \times 0.24 \times 0.18 \text{ mm}$

Data collection

Agilent Xcalibur (Eos, Gemini ultra)
diffractometer

Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator

Detector resolution: 16.0122 pixels mm^{-1}
 ω scans

Absorption correction: gaussian
(*CrysAlis PRO*; Agilent, 2013)

$T_{\min} = 0.755$, $T_{\max} = 0.821$

26858 measured reflections

4161 independent reflections

3394 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.037$

$\theta_{\max} = 32.5^\circ$, $\theta_{\min} = 3.4^\circ$

$h = -18 \rightarrow 17$

$k = -18 \rightarrow 12$

$l = -24 \rightarrow 21$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.037$

$wR(F^2) = 0.091$

$S = 1.04$

4161 reflections

130 parameters

0 restraints

Primary atom site location: heavy-atom method

Secondary atom site location: difference Fourier map

Hydrogen site location: constr

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0319P)^2 + 2.9104P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 1.21 \text{ e \AA}^{-3}$

$\Delta\rho_{\min} = -1.07 \text{ e \AA}^{-3}$

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) etc. and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

| | x | y | z | $U_{\text{iso}}^* / U_{\text{eq}}$ | Occ. (<1) |
|-----|--------------|--------------|---------------|------------------------------------|-----------|
| Co1 | 0.7500 | 0.2500 | 0.361342 (19) | 0.01381 (8) | |
| C1 | 0.86792 (13) | 0.37038 (14) | 0.36190 (10) | 0.0171 (3) | |
| C2 | 0.79973 (13) | 0.38033 (14) | 0.29066 (10) | 0.0169 (3) | |
| C3 | 0.68885 (14) | 0.39523 (14) | 0.31700 (11) | 0.0193 (3) | |
| C4 | 0.68838 (14) | 0.39480 (15) | 0.40471 (11) | 0.0206 (3) | |
| C5 | 0.79870 (14) | 0.37984 (14) | 0.43257 (10) | 0.0188 (3) | |

| | | | | | |
|------|--------------|--------------|--------------|--------------|-------------|
| C6 | 0.98932 (14) | 0.35672 (16) | 0.36341 (11) | 0.0221 (3) | |
| H6A | 1.0095 | 0.3102 | 0.4085 | 0.033* | |
| H6B | 1.0235 | 0.4270 | 0.3699 | 0.033* | |
| H6C | 1.0131 | 0.3242 | 0.3128 | 0.033* | |
| C7 | 0.83672 (15) | 0.38171 (16) | 0.20305 (11) | 0.0231 (4) | |
| H7A | 0.9052 | 0.3436 | 0.1984 | 0.035* | |
| H7B | 0.8459 | 0.4561 | 0.1853 | 0.035* | |
| H7C | 0.7828 | 0.3464 | 0.1693 | 0.035* | |
| C8 | 0.59249 (15) | 0.41374 (17) | 0.26198 (13) | 0.0268 (4) | |
| H8A | 0.5987 | 0.3678 | 0.2144 | 0.040* | |
| H8B | 0.5906 | 0.4891 | 0.2452 | 0.040* | |
| H8C | 0.5263 | 0.3962 | 0.2910 | 0.040* | |
| C9 | 0.59133 (16) | 0.41372 (19) | 0.45881 (14) | 0.0318 (4) | |
| H9A | 0.5256 | 0.3966 | 0.4292 | 0.048* | |
| H9B | 0.5896 | 0.4891 | 0.4756 | 0.048* | |
| H9C | 0.5965 | 0.3676 | 0.5064 | 0.048* | |
| C10 | 0.83534 (17) | 0.38163 (18) | 0.52022 (12) | 0.0281 (4) | |
| H10A | 0.7814 | 0.3462 | 0.5539 | 0.042* | |
| H10B | 0.8443 | 0.4561 | 0.5379 | 0.042* | |
| H10C | 0.9039 | 0.3437 | 0.5251 | 0.042* | |
| Co2 | 1.2500 | 0.2500 | 0.2500 | 0.01550 (10) | |
| Cl1 | 1.11315 (3) | 0.29420 (4) | 0.15956 (3) | 0.02050 (9) | |
| C11 | 1.2500 | 0.2500 | 0.5298 (2) | 0.0505 (9) | |
| H | 1.2150 | 0.1964 | 0.4946 | 0.061* | 0.3825 (19) |
| HA | 1.2850 | 0.3036 | 0.4946 | 0.061* | 0.3825 (19) |
| HB | 1.2469 | 0.1859 | 0.4946 | 0.061* | 0.1175 (19) |
| HC | 1.2531 | 0.3141 | 0.4946 | 0.061* | 0.1175 (19) |
| Cl2 | 1.35262 (12) | 0.1830 (2) | 0.58797 (8) | 0.0726 (5) | 0.765 (4) |
| Cl2A | 1.3635 (4) | 0.2446 (6) | 0.5851 (3) | 0.0726 (5) | 0.235 (4) |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|------|--------------|--------------|--------------|--------------|---------------|---------------|
| Co1 | 0.01106 (14) | 0.01787 (15) | 0.01251 (14) | 0.00152 (11) | 0.000 | 0.000 |
| C1 | 0.0155 (7) | 0.0193 (7) | 0.0167 (7) | -0.0010 (6) | -0.0006 (6) | -0.0008 (6) |
| C2 | 0.0162 (7) | 0.0183 (7) | 0.0163 (7) | 0.0004 (6) | -0.0001 (6) | 0.0012 (6) |
| C3 | 0.0172 (7) | 0.0186 (7) | 0.0221 (8) | 0.0032 (6) | -0.0017 (6) | -0.0003 (6) |
| C4 | 0.0186 (8) | 0.0208 (8) | 0.0224 (8) | 0.0032 (6) | 0.0015 (6) | -0.0042 (6) |
| C5 | 0.0177 (7) | 0.0217 (8) | 0.0171 (7) | -0.0002 (6) | 0.0007 (6) | -0.0042 (6) |
| C6 | 0.0151 (7) | 0.0317 (9) | 0.0195 (8) | -0.0029 (7) | -0.0005 (6) | 0.0007 (7) |
| C7 | 0.0219 (8) | 0.0304 (9) | 0.0169 (8) | -0.0021 (7) | 0.0005 (6) | 0.0031 (7) |
| C8 | 0.0191 (8) | 0.0298 (9) | 0.0316 (10) | 0.0068 (7) | -0.0050 (7) | 0.0037 (8) |
| C9 | 0.0223 (9) | 0.0390 (11) | 0.0342 (11) | 0.0054 (8) | 0.0083 (8) | -0.0124 (9) |
| C10 | 0.0272 (9) | 0.0399 (11) | 0.0171 (8) | -0.0027 (8) | -0.0007 (7) | -0.0060 (8) |
| Co2 | 0.01459 (13) | 0.01459 (13) | 0.0173 (2) | 0.000 | 0.000 | 0.000 |
| Cl1 | 0.01695 (18) | 0.0238 (2) | 0.02071 (18) | 0.00320 (14) | -0.00162 (14) | -0.00033 (15) |
| C11 | 0.056 (2) | 0.069 (3) | 0.0266 (16) | 0.000 (2) | 0.000 | 0.000 |
| Cl2 | 0.0681 (7) | 0.0970 (14) | 0.0527 (5) | 0.0388 (9) | -0.0021 (5) | 0.0055 (8) |
| Cl2A | 0.0681 (7) | 0.0970 (14) | 0.0527 (5) | 0.0388 (9) | -0.0021 (5) | 0.0055 (8) |

Geometric parameters (\AA , \circ)

| | | | |
|--------------------------------------|-------------|-------------------------|-------------|
| Co1—C1 ⁱ | 2.0576 (17) | C7—H7B | 0.9600 |
| Co1—C1 | 2.0576 (17) | C7—H7C | 0.9600 |
| Co1—C2 ⁱ | 2.0556 (17) | C8—H8A | 0.9600 |
| Co1—C2 | 2.0556 (17) | C8—H8B | 0.9600 |
| Co1—C3 | 2.0550 (17) | C8—H8C | 0.9600 |
| Co1—C3 ⁱ | 2.0550 (17) | C9—H9A | 0.9600 |
| Co1—C4 ⁱ | 2.0470 (17) | C9—H9B | 0.9600 |
| Co1—C4 | 2.0470 (17) | C9—H9C | 0.9600 |
| Co1—C5 ⁱ | 2.0521 (17) | C10—H10A | 0.9600 |
| Co1—C5 | 2.0522 (17) | C10—H10B | 0.9600 |
| C1—C2 | 1.433 (2) | C10—H10C | 0.9600 |
| C1—C5 | 1.432 (2) | Co2—Cl1 ⁱⁱ | 2.2915 (4) |
| C1—C6 | 1.492 (2) | Co2—Cl1 ⁱⁱⁱ | 2.2915 (4) |
| C2—C3 | 1.432 (2) | Co2—Cl1 | 2.2915 (4) |
| C2—C7 | 1.496 (2) | Co2—Cl1 ^{iv} | 2.2915 (4) |
| C3—C4 | 1.428 (3) | C11—H | 0.9700 |
| C3—C8 | 1.496 (2) | C11—HA | 0.9700 |
| C4—C5 | 1.433 (2) | C11—HB | 0.9700 |
| C4—C9 | 1.494 (3) | C11—HC | 0.9700 |
| C5—C10 | 1.496 (3) | C11—Cl2 ⁱⁱⁱ | 1.771 (2) |
| C6—H6A | 0.9600 | C11—Cl2 | 1.771 (2) |
| C6—H6B | 0.9600 | C11—Cl2A ⁱⁱⁱ | 1.654 (5) |
| C6—H6C | 0.9600 | C11—Cl2A | 1.654 (5) |
| C7—H7A | 0.9600 | | |
| | | | |
| C1—Co1—C1 ⁱ | 179.49 (9) | C9—C4—Co1 | 128.93 (14) |
| C2—Co1—C1 ⁱ | 139.66 (7) | C1—C5—Co1 | 69.81 (9) |
| C2 ⁱ —Co1—C1 ⁱ | 40.78 (6) | C1—C5—C4 | 108.10 (15) |
| C2—Co1—C1 | 40.78 (6) | C1—C5—C10 | 126.23 (16) |
| C2 ⁱ —Co1—C1 | 139.66 (7) | C4—C5—Co1 | 69.34 (10) |
| C2—Co1—C2 ⁱ | 111.91 (9) | C4—C5—C10 | 125.54 (16) |
| C3—Co1—C1 ⁱ | 111.33 (7) | C10—C5—Co1 | 129.57 (14) |
| C3 ⁱ —Co1—C1 ⁱ | 68.86 (7) | C1—C6—H6A | 109.5 |
| C3 ⁱ —Co1—C1 | 111.33 (7) | C1—C6—H6B | 109.5 |
| C3—Co1—C1 | 68.86 (7) | C1—C6—H6C | 109.5 |
| C3 ⁱ —Co1—C2 | 111.35 (7) | H6A—C6—H6B | 109.5 |
| C3—Co1—C2 | 40.77 (7) | H6A—C6—H6C | 109.5 |
| C3—Co1—C2 ⁱ | 111.35 (7) | H6B—C6—H6C | 109.5 |
| C3 ⁱ —Co1—C2 ⁱ | 40.77 (7) | C2—C7—H7A | 109.5 |
| C3—Co1—C3 ⁱ | 138.87 (10) | C2—C7—H7B | 109.5 |
| C4 ⁱ —Co1—C1 | 110.99 (7) | C2—C7—H7C | 109.5 |
| C4—Co1—C1 | 68.82 (7) | H7A—C7—H7B | 109.5 |
| C4—Co1—C1 ⁱ | 110.99 (7) | H7A—C7—H7C | 109.5 |
| C4 ⁱ —Co1—C1 ⁱ | 68.82 (7) | H7B—C7—H7C | 109.5 |
| C4 ⁱ —Co1—C2 ⁱ | 68.47 (7) | C3—C8—H8A | 109.5 |
| C4 ⁱ —Co1—C2 | 138.86 (7) | C3—C8—H8B | 109.5 |
| C4—Co1—C2 | 68.47 (7) | C3—C8—H8C | 109.5 |
| C4—Co1—C2 ⁱ | 138.86 (7) | H8A—C8—H8B | 109.5 |

| | | | |
|--------------------------------------|-------------|---|--------------|
| C4 ⁱ —Co1—C3 | 179.56 (8) | H8A—C8—H8C | 109.5 |
| C4—Co1—C3 | 40.75 (7) | H8B—C8—H8C | 109.5 |
| C4 ⁱ —Co1—C3 ⁱ | 40.74 (7) | C4—C9—H9A | 109.5 |
| C4—Co1—C3 ⁱ | 179.56 (8) | C4—C9—H9B | 109.5 |
| C4 ⁱ —Co1—C4 | 139.65 (11) | C4—C9—H9C | 109.5 |
| C4 ⁱ —Co1—C5 ⁱ | 40.92 (7) | H9A—C9—H9B | 109.5 |
| C4 ⁱ —Co1—C5 | 111.46 (7) | H9A—C9—H9C | 109.5 |
| C4—Co1—C5 ⁱ | 111.46 (7) | H9B—C9—H9C | 109.5 |
| C4—Co1—C5 | 40.92 (7) | C5—C10—H10A | 109.5 |
| C5—Co1—C1 | 40.79 (7) | C5—C10—H10B | 109.5 |
| C5 ⁱ —Co1—C1 | 138.77 (7) | C5—C10—H10C | 109.5 |
| C5 ⁱ —Co1—C1 ⁱ | 40.79 (7) | H10A—C10—H10B | 109.5 |
| C5—Co1—C1 ⁱ | 138.77 (7) | H10A—C10—H10C | 109.5 |
| C5—Co1—C2 ⁱ | 179.55 (7) | H10B—C10—H10C | 109.5 |
| C5 ⁱ —Co1—C2 ⁱ | 68.45 (7) | C11 ⁱⁱⁱ —Co2—Cl1 ⁱⁱ | 114.385 (12) |
| C5 ⁱ —Co1—C2 | 179.55 (7) | C11 ⁱⁱⁱ —Co2—Cl1 | 100.04 (2) |
| C5—Co1—C2 | 68.45 (7) | C11 ⁱⁱ —Co2—Cl1 | 114.385 (11) |
| C5 ⁱ —Co1—C3 | 139.45 (7) | C11 ⁱⁱⁱ —Co2—Cl1 ^{iv} | 114.386 (11) |
| C5—Co1—C3 | 68.73 (7) | C11 ⁱⁱ —Co2—Cl1 ^{iv} | 100.04 (2) |
| C5—Co1—C3 ⁱ | 139.45 (7) | C11—Co2—Cl1 ^{iv} | 114.385 (11) |
| C5 ⁱ —Co1—C3 ⁱ | 68.72 (7) | H—C11—HA | 107.5 |
| C5 ⁱ —Co1—C5 | 111.19 (10) | H—C11—HC | 102.2 |
| C2—C1—Co1 | 69.54 (9) | HA—C11—HB | 102.2 |
| C2—C1—C6 | 126.87 (15) | HB—C11—HC | 107.6 |
| C5—C1—Co1 | 69.40 (10) | Cl2 ⁱⁱⁱ —C11—H | 108.4 |
| C5—C1—C2 | 107.49 (14) | Cl2—C11—H | 108.4 |
| C5—C1—C6 | 125.60 (15) | Cl2 ⁱⁱⁱ —C11—HA | 108.4 |
| C6—C1—Co1 | 127.98 (13) | Cl2—C11—HA | 108.4 |
| C1—C2—Co1 | 69.69 (9) | Cl2 ⁱⁱⁱ —C11—HB | 131.4 |
| C1—C2—C7 | 126.67 (15) | Cl2—C11—HB | 88.3 |
| C3—C2—Co1 | 69.59 (10) | Cl2 ⁱⁱⁱ —C11—HC | 88.3 |
| C3—C2—C1 | 108.52 (14) | Cl2—C11—HC | 131.4 |
| C3—C2—C7 | 124.72 (15) | Cl2—C11—Cl2 ⁱⁱⁱ | 115.3 (2) |
| C7—C2—Co1 | 129.17 (13) | Cl2A ⁱⁱⁱ —C11—H | 88.8 |
| C2—C3—Co1 | 69.64 (9) | Cl2A—C11—H | 131.6 |
| C2—C3—C8 | 125.70 (16) | Cl2A ⁱⁱⁱ —C11—HA | 131.6 |
| C4—C3—Co1 | 69.33 (10) | Cl2A—C11—HA | 88.8 |
| C4—C3—C2 | 107.63 (15) | Cl2A—C11—HB | 108.7 |
| C4—C3—C8 | 126.60 (16) | Cl2A ⁱⁱⁱ —C11—HB | 108.7 |
| C8—C3—Co1 | 128.79 (13) | Cl2A—C11—HC | 108.7 |
| C3—C4—Co1 | 69.93 (10) | Cl2A ⁱⁱⁱ —C11—HC | 108.7 |
| C3—C4—C5 | 108.25 (15) | Cl2A—C11—Cl2 ⁱⁱⁱ | 108.7 (2) |
| C3—C4—C9 | 126.30 (17) | Cl2A ⁱⁱⁱ —C11—Cl2 | 108.7 (2) |
| C5—C4—Co1 | 69.73 (10) | Cl2A—C11—Cl2A ⁱⁱⁱ | 114.1 (4) |
| C5—C4—C9 | 125.35 (17) | | |

Symmetry codes: (i) $-x+3/2, -y+1/2, z$; (ii) $y+1, -x+3/2, -z+1/2$; (iii) $-x+5/2, -y+1/2, z$; (iv) $-y+3/2, x-1, -z+1/2$.

Hydrogen-bond geometry (\AA , $^\circ$)

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|----------------------------|--------------|--------------------|-------------|----------------------|
| C11—H···Cl1 ^{iv} | 0.97 | 2.71 | 3.548 (3) | 145 |
| C11—HA···Cl1 ⁱⁱ | 0.97 | 2.71 | 3.548 (3) | 145 |

Symmetry codes: (ii) $y+1, -x+3/2, -z+1/2$; (iv) $-y+3/2, x-1, -z+1/2$.